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Assessment of Carbonation Potential in Lime-treated Clayey Soil based on Thermogravimetric and Fourier Transform Infrared Spectroscopy Analyses

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ABSTRACT

Lime treatment is a widely adopted technique to improve the workability and mechanical performance of clayey soils comprising pavement subgrade, embankments or building foundations. Although it is a widespread technique, the interaction of atmospheric and soil CO_2 with the calcium-bearing phases of the treated soil is not sufficiently explored. The detailed chemical characterisation of lime-treated soils helps evaluate their potential to carbonate during different stages of curing. The present study investigates the carbonation potential of lime-treated and compacted clayey soil cured for up to 365 days. After the proposed curing periods, the samples were exposed to 7 days of carbonation in a specially fabricated closed cell, which maintains a 3% CO_2 concentration. The unconfined compressive strength (UCS) test was adopted to study the mechanical characteristics. The chemical analyses were performed using thermogravimetric analysis (TGA) and Fourier-transform infrared (FTIR) spectroscopy. The results depicted an average 30% reduction in the unconfined compressive strength and a 21% reduction in the elastic modulus upon carbonation in lime-treated soils. The analytical techniques identified the presence of carbonation of lime and hydrated phases at each curing period. The reduced engineering performance upon carbonation is attributed to the combined effects of lime paucity for pozzolanic reactions and carbonation of cementitious gels.

Keywords: Carbonation, lime stabilisation, strength, stiffness, TGA, FTIR

1 INTRODUCTION

Lime stabilisation of clayey soils encountered in the field is a preferred method owing to its simplicity in design and execution procedure as well as cost-effectiveness (Cherian et al., 2018; Mishra et al., 2019). Adopting this technique reduces the total construction time and the wastage arising from alternative methods like soil replacement. In the field, lime stabilisation is executed by a set of sequential processes involving (i) scarification and pulverisation of virgin soil, (ii) lime spreading, (iii) preliminary mixing and watering, (iv) compaction, (v) and moist curing. When lime is introduced into the clayey soil, it dissolves in the pore fluid to Ca^{2+} ions and replaces the monovalent ions on the clay surface. As a result, the thickness of diffused double layer reduces, and the soil transforms into a flocculated and coarse-grained material. This process is called soil modification and is adopted to make the soil more workable, which expedites the execution of construction activities (Rogers et al., 1997). However, additional lime is required to attain the desired strength characteristics of a subgrade beneath pavement or underlying bearing strata beneath the superstructures. The improvement in the strength is achieved via pozzolanic reactions between lime and silica-alumina ions dissolved from the clays at alkaline pH conditions. These reactions progress slowly and take place throughout the service period of the treated layer as long as lime is available. The cementitious gels like calcium silicate hydrate (CSH) and calcium silicate aluminate hydrate (CASH) precipitated from this reaction bind the soil aggregates and increase the compressive strength. The stability of these reaction products and the availability of residual lime for sustained pozzolanic reactions help in the overall durability of lime-treated soil layers (Chakraborty & Nair, 2018). Several studies were conducted to assess the durability of lime-treated soil by conducting wetting and drying cycles as per ASTM D559, (2016). The reversal of plasticity properties and reduction

in strength was commonly observed by a few researchers, whereas others suggested the protocol to be severe and did not replicate the climatic conditions in the field (Padmaraj & Arnepalli, 2021a). Also, an in-depth investigation was not made regarding the chemical reactions that occur in the field during the service period of lime-treated soil layers.

Along with cation exchange and pozzolanic reactions, carbonation of calcium-bearing phases like lime and cementitious phases takes place. In the field, exposure of the lime-treated layer to CO_2 takes place either during the construction time or service period. During the mixing and pulverisation stage, as explained above, the treated soil is exposed to atmospheric air containing CO_2. Whereas, after construction and sealing of the treated layer, CO_2 present, if any, in the soil can contribute to the reaction. The source of carbonation in soils arises primarily from the decomposition of organic matter, which release CO_2. As per Rezagholilou & Nikraz, (2013), an organic content of 5% is sufficient to release 3% CO_2 into the soil air. The penetration of atmospheric air through surficial cracks also creates the possibility for carbonation in a lime-treated layer. The reaction is considered deleterious due to the consumption of lime which limits the clay-lime reactions in the treated soils. Also, CO_2 interacts with calcium in the cementitious phases and reduces its stability and, consequently, its cohesive property. The reduction in the apparent cohesion imparted by these phases affects the mechanical performance of the compacted layer.

Many laboratory and field case studies reported carbonation reactions in the lime and cement- treated layers. Netterberg & Paige-Green, (1984) reported carbonation to be the primary cause of surfacing failure and disintegration of the upper base course of several layers of pavement in South Africa. Bagonza et al., (1987) reported an average 50% reduction in compressive strength when lime-treated soil was exposed to CO_2-rich air. According to the researchers, an environment that has a higher concentration of CO_2, higher temperature, or relatively high humidity are favourable conditions for carbonation. Bredenkamp & Scullion, (1995) observed carbonation in the cemented base layer of a state highway in the United States, which resulted in cracking and a reduction in strength. Similarly, Yeo & Nikraz, (2012) identified carbonation as a cause for observed curvature, deflection, and rutting of certain sections of the cement-treated base of trial sections of Reid Highway in Western Australia. On the contrary, field studies by Haas & Ritter, (2019) on lime-treated embankments identified 37% carbonation with a limited reduction in engineering performance. Similar observations were noted by Das et al., (2021) by exposing lime-treated embankment to atmospheric carbonation.

Given contradicting field observations related to the carbonation of lime-treated soil layers, it is necessary to conduct a detailed investigation of the effects of carbonation, considering a multiscale analysis involving both macro and micro-level changes. Few laboratory studies on lime-treated soils reported calcite precipitation from mineralogical analysis and microscopic imaging (Lemaire et al., 2013; Al-Mukhtar et al., 2012). Bandipally et al., (2018) observed retardation of pozzolanic reaction due to lime carbonation, whereas Xu et al., (2020) noticed partial recovery of the expansive potential of lime-treated expansive soil. Further, a study by Vitale et al., (2021) observed reduced strength for lime-treated kaolinitic soil exposed to the atmosphere compared to the specimens cured under a sealed environment. These studies focused on the consequence of carbonation; however, limited studies attempted a detailed chemical characterisation of the treated soil to explore the mechanism and effects of carbonation on the engineering performance of lime-treated soil.

TGA (Thermogravimetric analysis) and FTIR (Fourier transform infrared) spectroscopy are commonly used analytical techniques to study the thermal behaviour and chemical structure of materials. Recently, these tools have been widely adopted to study the time-dependent changes in the chemistry of clayey soils upon stabilisation (Saeed et al., 2015; Bandipally et al., 2018). The technique is also used in cement concrete research to evaluate the effects of carbonation (Qiu, 2020). However, limited studies have been conducted on the carbonation behaviour in stabilised soils. Given this, the present study investigates the carbonation potential and consequent changes in the compressive strength and elastic modulus of compacted lime-treated soil by exposing the samples to CO_2. Further, the role of initial lime content (4% and 8%) and curing ages (7, 90, 180 and 365 days) of treated soil on the effect of carbonation is studied by conducting chemical analysis using TGA and FTIR spectroscopy analyses.

2 METHODOLOGY

2.1 Material characterisation

The soil was sampled from a lakebed within the premises of the Indian Institute of Technology Madras, Chennai. Further, the physical, chemical, geotechnical, and mineralogical properties were assessed following the ASTM guidelines (Table 1). The selected soil consists of 55% of clay fraction, 41% silt and 4% sand and a plasticity index of 18%. From the measured grain size distribution and consistency limits, the soil was classified as clays of low compressibility. The mineralogical properties revealed the dominant mineral as illite with traces of quartz and feldspar. Additional non-clay minerals contribute to excess silica content, increasing the reactivity with lime. Calcium hydroxide with 96% purity, measured from XRF analysis, was used as the additive in the study.

Table 1. *Characterisation of selected soil*

Property	Value
Specific gravity (-) (ASTM D5550, 2014)	2.62
pH (-) (ASTM D4972, 2019)	7.8
Cation exchange capacity (cmol (+)·kg^{-1}) (ASTM D7503, 2018)	87.1
Plasticity index (%) (ASTM D4318-17e1, 2018)	18
Clay-size fraction (%) (ASTM D7928, 2021)	55
USCS classification (ASTM D2487, 2020)	CL

2.2 Sample preparation and curing

The excavated soil was pulverised, dried and sieved through 1 mm before conducting accelerated carbonation experiments. The powdered soil was then mixed with water equivalent to the plastic limit and kept in airtight bags for 48 hours. This procedure enabled the equilibration of moisture to simulate the condition in the field. Further, lime content was added in required proportions (4% and 8% by dry weight), and respective cylindrical-shaped samples with 38 mm diameter and 76 mm height were prepared as per standard Proctor compaction protocol (ASTM D698, 2021). These composites were then sealed using aluminium foil and cured at ambient temperature of 27°C and 95% relative humidity for up to 365 days.

2.3 Carbonation experiments

For carbonation experiments, 3% v/v CO_2 was used as the respective value mimics the mechanism of atmospheric carbonation (Castellote et al., 2008). Also, it closely represents the subsoil concentration of CO_2 (Netterberg & Paige-green et al., 1984). The selected gas mixture was manually mixed before carbonation experiments. After each curing period, the specimens were placed in a closed cell (Figure 1), and the CO_2-rich gas was purged for fixed time intervals, and the procedure was continued for 7 consecutive days. Further, the specimens were taken out to assess the mechanical and micro-level characteristics. As a control, samples purged with nitrogen gas were used. They are mentioned as non-carbonated samples in the following sections.

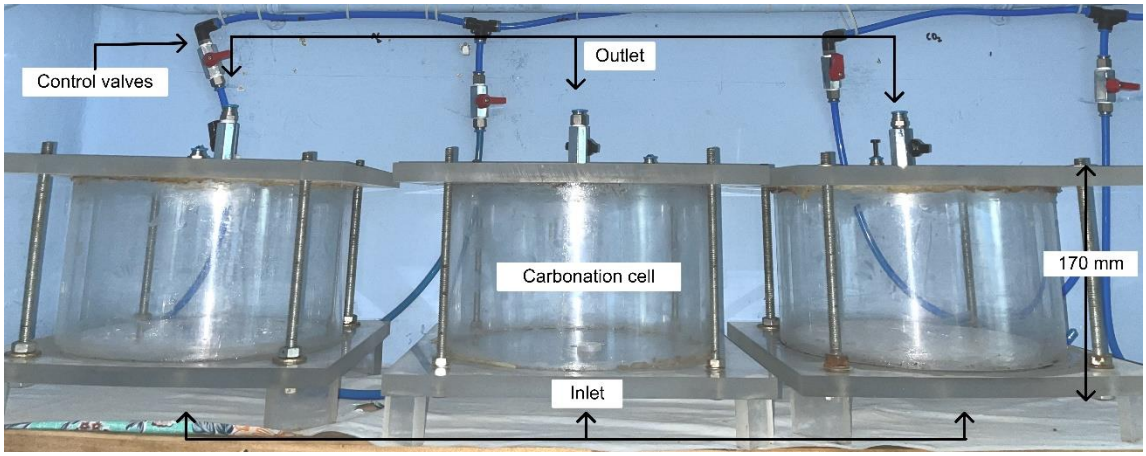


Figure 1. *Arrangement of closed cells for carbonation experiments*

2.4 Mechanical tests

Lime treatment is predominantly used to stabilise the subgrade or subbase layers of the pavement. The long-term strength and stiffness of the pavements under heavy traffic loads are important performance criteria used extensively in the field. In this study, the potential of lime-treated soil to resist the effect of carbonation on compressive strength performance was assessed using unconfined compressive strength (UCS) tests. The specimens were loaded at a rate of 0.5 mm/minute until failure. The ultimate strength was then estimated from the stress-strain curves obtained in triplicate for both carbonated and non-carbonated specimens. Further, the secant modulus (E_{sec}) was determined based on the relationship between the measured values of axial stress and deformation response. It was estimated as the ratio of 50% of ultimate stress to the corresponding axial strain. After failure, the compacted samples were powdered prior to conducting micro-level chemical tests (Figure 2).

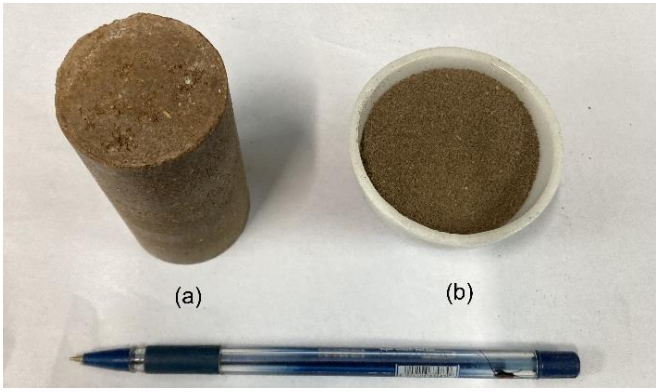


Figure 2. *Pictorial representation of (a) compacted sample for UCS tests and (b) powdered sample after UCS test for chemical analysis*

2.5 Thermogravimetric analysis

To calculate the amount of calcium carbonate and hydrates present, thermogravimetric analysis was conducted by employing Discovery SDT650 (TA Instruments, USA). 15-20 mg of representative samples were powdered and freeze-dried under a vacuum to prevent carbonation during the sample preparation. Following, it was heated at a controlled rate of 10ºC/min from 30ºC to 1000ºC under a non-reactive N_2 gas environment. The observed weight curve and its derivative were plotted against temperature. The derivative of the weight loss curve (DTG) was further analysed to check the weight losses occurring across the decomposition regime of hydrates and carbonate present in the lime-treated soil.

2.6 Fourier Transform Infrared spectroscopy

The infrared (IR) spectra of lime-treated soil exposed to carbonated and non-carbonated conditions were obtained using attenuated total reflectance (ATR) mode of analysis, with the aid of a bench-top Nicolet IS-50 FTIR spectrometer (Thermo Fischer Scientific, USA), equipped with an IR source, an extended potassium bromide beamsplitter, and a deuterated triglycine sulfate (DTGS) detector. The working conditions involved atmospheric air, dual beam IR data type, and spectral range of 600–4000 cm^{-1}. For each sample, 128 accumulations were recorded with a resolution of 4 cm^{-1}. The obtained spectrum consisted of wavenumber (cm^{-1}) as the abscissa and absorbance as the ordinate. Before analysis, the spectra were subjected to baseline and advanced ATR corrections.

3 RESULTS AND DISCUSSION

3.1 Variation in the strength and elastic modulus in lime-treated soil

The UCS and E_{sec} of virgin soil were estimated to be 0.23 MPa and 5.67 MPa respectively. Comparing the strength and modulus of non-carbonated specimens with curing age and lime content (Table 1), it is inferred that stabilisation is effective as lime induced considerable improvement in the engineering properties of soil. In addition to the reaction with clay minerals, non-clay minerals in the virgin soil

supplied additional silica, which enhanced the rate of pozzolanic reactions (Cherian et al., 2018). The cementitious gels precipitated after the pozzolanic reaction bonded the modified clayey aggregates and enhanced the strength and stiffness of the composite.

***Table 2**. Mechanical characteristics of treated soil after carbonation*

Lime content (%)	Curing age before carbonation (days)	Unconfined compressive strength (MPa)		Elastic modulus, E_{sec} (MPa)	
		Non-carbonated	Carbonated	Non-carbonated	Carbonated
4	7	1.3	0.7	36.4	26.5
	90	2.4	1.4	46.7	39.1
	180	2.5	1.8	102.8	69.3
	365	1.9	1.4	68.9	36.9
8	7	1.7	1.1	60.7	60.7
	90	4.0	3.0	106.6	85.8
	180	4.6	3.5	146.5	126.3
	365	3.9	3.3	154.8	111.2

Further, the effect of carbonation is assessed by comparing the compressive strength of carbonated specimens with that of corresponding non-carbonated specimens. On average, a 30% reduction in the compressive strength and a 21% reduction in the elastic modulus were observed after exposing the specimens to carbonation. It showed that the carbonation reaction in lime-treated soil reduced the beneficial effects brought by lime. In the lime-treated soil, CO_2 reacts with lime and cementitious phases to precipitate calcium carbonate. The precipitation of calcium carbonate induces tensile stress, which can partly cause microcracking (Netterberg & Paige-Green, 1984). Further, the carbonation of cementitious gels reduces their Ca/Si ratio, which affects their cohesive property (Ashraf, 2016). These two changes bring significant changes to the engineering performance of soils which are stabilised with lime.

The variation in the UCS and stiffness upon carbonation is presented in Figure 3. Comparatively, 4% lime-treated soils exhibited a significant reduction in the strength and elastic modulus values. As lime content increased, the rate of precipitation of cementitious phases increased, which could have created a tortuous pathway for CO_2 permeation (Barmade et al., 2022). Also, the improved stability of cementitious phases with adding extra lime, as reported by Chakraborty & Nair, (2018), also contributes towards increasing resistance to carbonation reactions. As the curing age of the specimens increased, the percentage reduction in the compressive strength due to CO_2 exposure diminished. Lime-treated soils undergo continuous pozzolanic reactions and precipitate cementitious phases during curing. The pore refinement that occurs with the precipitation of these phases during curing may reduce the extent of CO_2 permeation and the associated deleterious effects of carbonation.

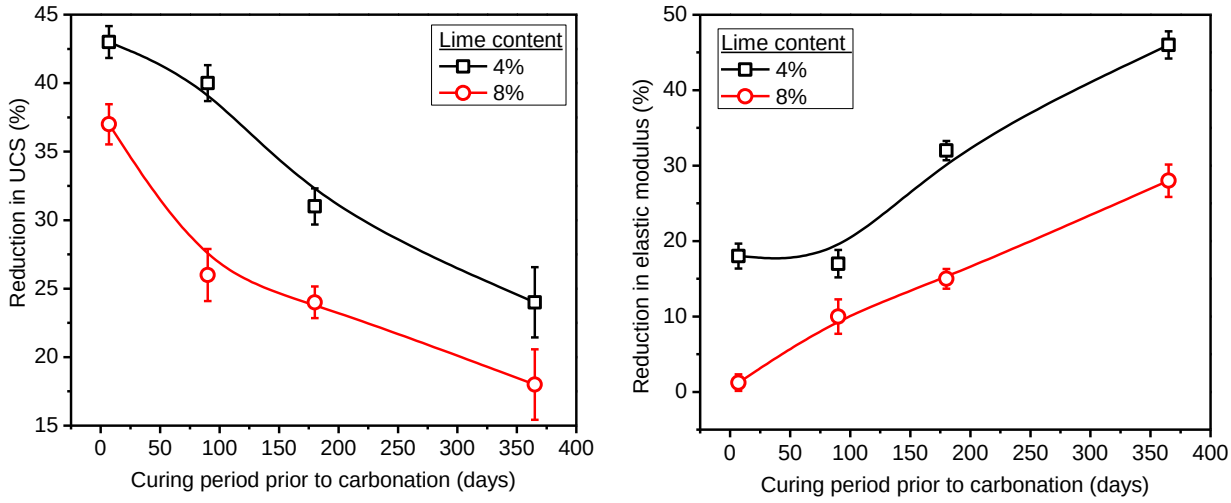


***Figure 3.** Percentage reduction in the UCS (left) and stiffness (right) upon carbonation in lime-treated soil*

On the other hand, the percentage reduction in the stiffness after carbonation was predominant in the specimens with longer curing ages (Figure 3). Lime carbonation occurs in the short-term cured samples due to the excess amount of unreacted lime. Lime carbonation mainly results in pore-filling by carbonates, which partially increases the stiffness, whereas carbonation of CSH reduces the cohesiveness of the matrix, which may result in pore coarsening and reduction in the stiffness (Deneele et al., 2021). The combined influence of these two chemical reactions, the degree of which varies with curing age, affects the variation in the strength and stiffness of lime-treated soils. For improved clarity on the reaction processes, chemical analyses were conducted.

3.2 Chemical modifications upon carbonation

The variations in the chemical composition of treated soils under carbonated and non-carbonated exposure conditions were evaluated by conducting a thermal analysis. Figure 4 shows the thermogravimetric curves obtained for lime (8%) treated soils. The major constituents of interest in the soil-lime composite were residual lime, hydrates and carbonates. In the carbonate decomposition region, two peaks were observed, a broader and sharp peak with a decomposition temperature range of 550 ºC - 600 ºC and 600 ºC - 710ºC, respectively. Previous studies reported carbonate peaks with lower decomposition temperatures to have emerged from amorphous carbonates that evolved from cementitious phases, whereas sharp peaks from crystalline carbonates emerged from residual lime (Villain et al., 2007; Padmaraj & Arnepalli, 2021b). Given this, it can be assumed that the relative percentage of lime and cementitious phases present in the composite determine the nature of carbonates formed in treated soil.

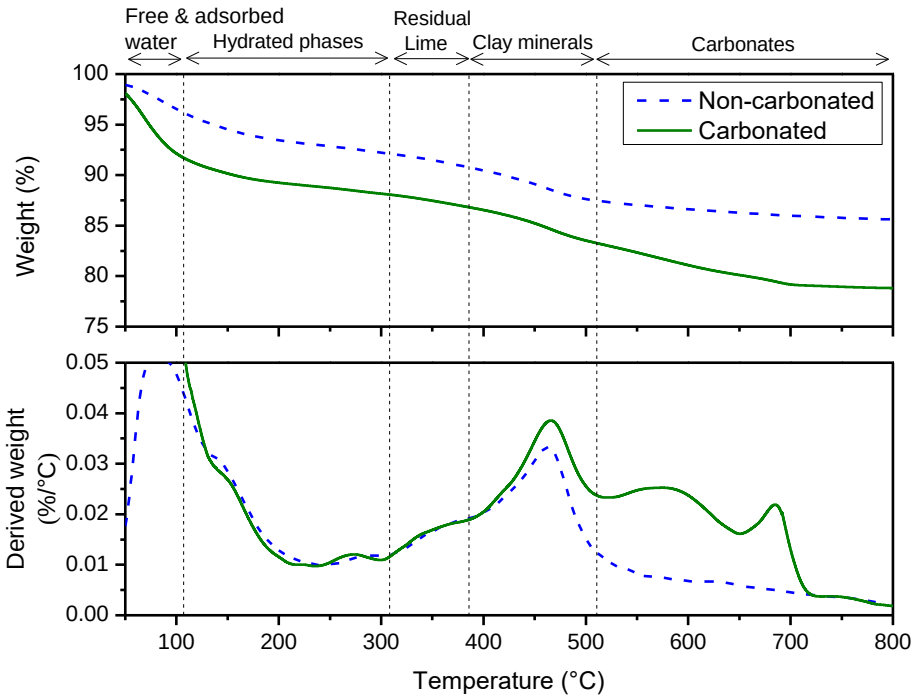


***Figure 4**. Thermogravimetric (TG) and its derivative (DTG) curve of carbonated 8% lime-treated soil initially cured up to* 90 days

As shown in Figure 5, the mass loss due to lime decomposition reduced, whereas the mass loss due to dehydroxylation of hydrated phases increased with curing age. It shows the consumption of lime by pozzolanic materials in the soil to form hydrated phases like CSH. Hence, it is understood that CO_2 interaction is primarily with lime in the initial curing ages, whereas as curing progresses, the reaction is mainly with the cementitious gels. The cementitious gels undergo carbonation by the removal of calcium from their chemical structure by a process called decalcification. The reduction in the cementitious gels with carbonation is also observed. As a part of this process, these phases partially lose their ability to bind the aggregates and hence reduce the strength of the lime-treated soil, as observed in Figure 3.

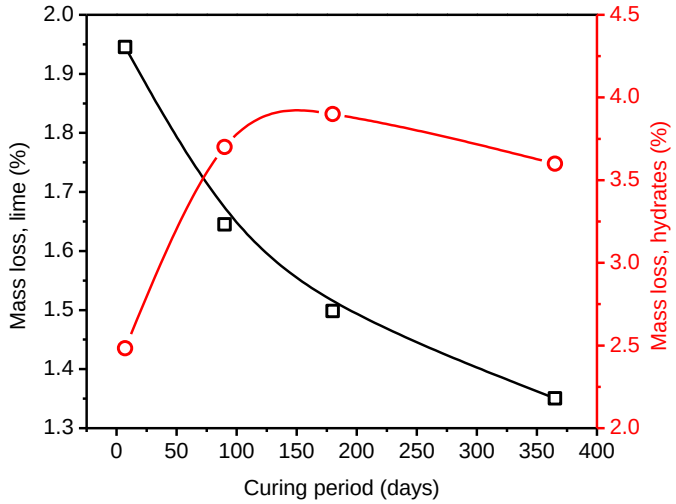


Figure 5. *Variation in the hydrates and residual lime in non-carbonated 8% lime-treated samples*

Further, mass loss corresponding to the decomposition of carbonates that are formed after exposure to CO_2 was measured from the TG curve. Figure 6 shows the estimated values of mass loss of carbonates formed in the treated soil with varying initial curing periods before carbonation. The results depicted a decreasing trend of estimated carbonates with the curing period. A similar trend is observed for two lime contents adopted for the study. It showed that as the curing period of the lime-treated samples increased, they became more resistant to carbonation. The observed results can be attributed to the changes in the chemical composition and pore structure that takes place with curing in lime-treated soils. As reported in Figure 5, the cementitious phases are predominant in the long-term cured samples. The carbonation reaction of these phases is not as quick as free lime. Upon lime stabilisation, the macropore develops initially, but later they are filled by the hydrated products of lime-soil reactions. The precipitation of cementitious phases in the mixture reforms the compacted soil to increase the density with a reduced volume of macropores. As gas permeation mainly takes place through the macropores, the extent of carbonation reduces as the curing period of the samples increases. The transformation of the pore structure of the treated soil affect the extent of gas diffusion, whereas the pozzolanic reaction between clay and lime affects the amount of carbonate precipitation.

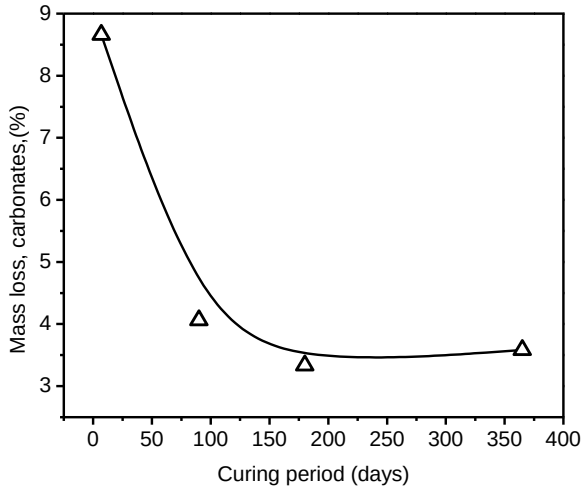


Figure 6. *Variation in the carbonates formed in the 8% lime-treated soil with varying initial curing periods*

Figure 7 presents the FTIR spectra obtained in ATR mode for both carbonated and non-carbonated samples with different curing ages before exposure to CO_2. The spectra comprised two regions: a functional group region in the range of 4000 cm^{-1} and 1450 cm^{-1}, consisting of structural hydroxyls, and a fingerprint region in the range of 1450 cm^{-1} to 500 cm^{-1} consisting of carbonate and silica-alumina bonds (Madejova & Komadel, 2001). The changes in the intensity of absorbance bands corresponding

to carbonates, hydroxyls, water, silica, and alumina represents the nature of changes in the chemical structure during pozzolanic and reactions.

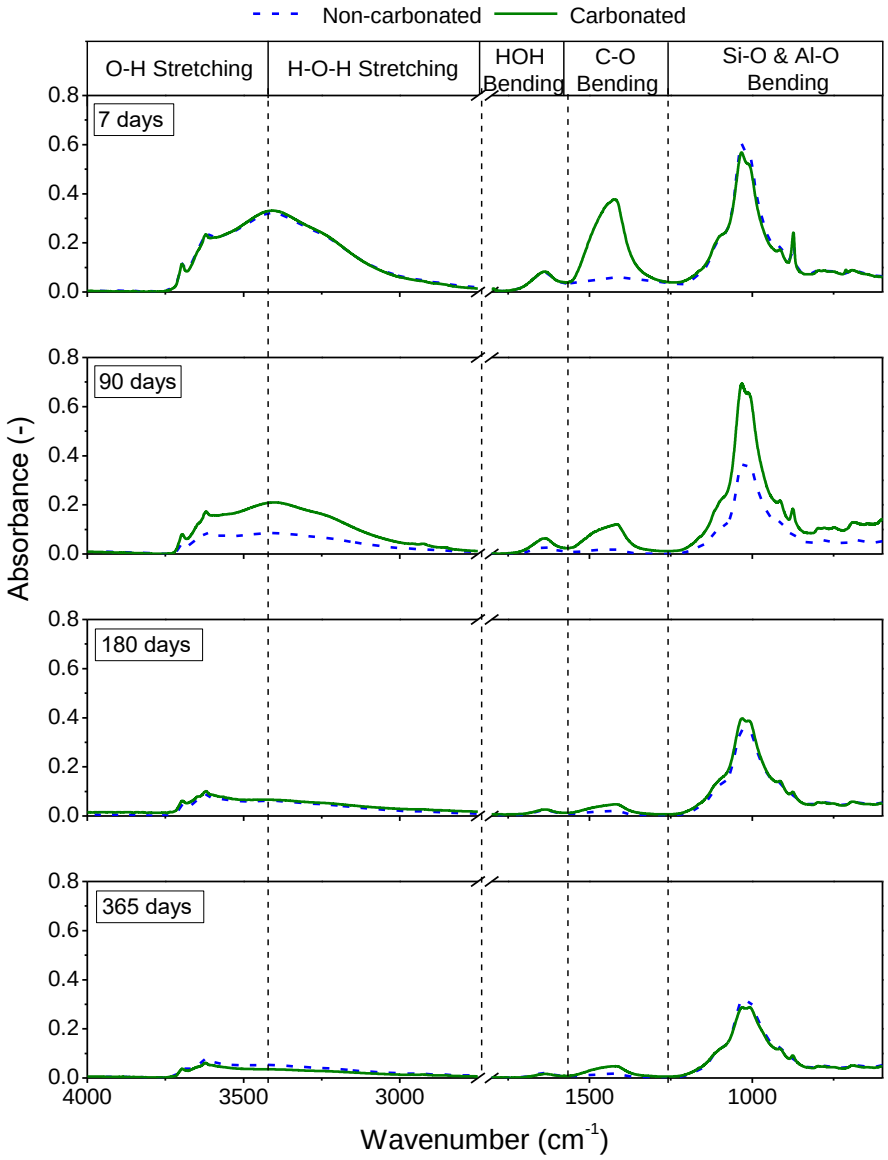


Figure 7. *FTIR spectra of non-carbonated and carbonated 8% lime-treated soils*

The broad peak at 3420 cm^{-1} depicts the stretching bonds of water and is related to the adsorbed water, whereas the sharp peak at 1635 cm^{-1} is related to the bending modes of water (Padmaraj & Arnepalli, 2022). Reduction in the absorbance of these bonds with curing age is observed in the cured samples. The results can be related to the diminishing water-holding capacity of clays due to the chemical modifications brought by lime. As lime interacts with clays, cation exchange reactions induces a reduction in the thickness of diffused double layer, which consequently reduces the water-holding capacity of the soil (Dash & Hussain, 2012). Similarly, a considerable reduction in the intensities of Si-O and Al-O bonds in the fingerprint region is noted. The dissolution of silica and alumina from the clays for pozzolanic reactions reduces their presence in the chemical structure of clay as observed by Bandipally et al., (2018). These observations show that there are significant alterations to the structure of clays due to lime stabilisation.

For carbonated soil lime composites, the peaks corresponding to carbonates were observed at 1420 cm^{-1} and 875 cm^{-1}corresponding to stretching and bending modes of vibration (Smidt & Meissl, 2007; Padmaraj & Arnepalli, 2022). The intensities of carbonate peaks were relatively less for long-term cured samples. The observation corroborated with TGA results, indicating the enhanced resistance offered by long-term cured samples against carbonation. Further, a slight increase in the intensity of the Si-O bond

at 1030 cm^{-1} is noted, especially for specimens with 90 days of curing age. Similar observations are noted by Wu & Ye (2016) on cement mortar after carbonation and have related this phenomenon to silica polymerisation that takes place after the carbonation of cementitious phases like CSH. Assuming that 90-days cured samples have sufficient macropores for gas permeation and unstable or newly developed cementitious phases which can be easily carbonated, this phenomenon could be associated to the effects of CSH carbonation. However, further studies are required to validate the inference derived from these observations.

4 CONCLUSIONS

Carbonation is a chemical reaction that proves to be detrimental to the performance of lime-treated soil. It not only consumes lime which is required for pozzolanic reactions, but also carbonates the cementitious phases, which affect the strength and stiffness of the treated soil. The significant findings observed from the present study are presented below.

- On average, a 30% reduction in the strength and a 21% reduction in the elastic modulus is observed for lime-treated samples upon carbonation
- The reduction in the engineering performance is attributed mainly to the carbonation of cementitious phases and partly to the tensile stresses induced by the precipitation of calcium carbonates
- Carbonation-induced reduction in the strength predominantly occurred in the short-term cured specimens, whereas a higher reduction in the stiffness occurred in the long-term cured specimens
- The relative percentage of lime and cementitious phases in treated soil varied with curing age due to lime consumption and pozzolanic reactions
- Lime carbonation is the predominant reaction in the initial curing ages, whereas CSH carbonation occurs in the long-term
- Carbonation resistance improved with curing age in lime-treated soils

The findings of the present study emphasise the containment of lime-treated soil layers up to 90 days after construction by proper barrier systems to prevent carbonation and subsequent reduction in the engineering performance. Also, the deleterious effects of carbonation reduced drastically upon the addition of extra lime content. Hence, it is advised to revise the design methodology by incorporating additional lime content to make the lime-treated soil more resistant to carbonation.

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